



PCT/US 04/41372



INVESTOR IN PEOPLE

PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

REC'D 17 JAN 2005

WIPO

PCT

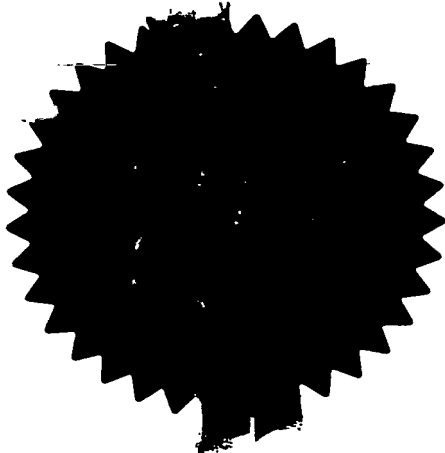
The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



Signed

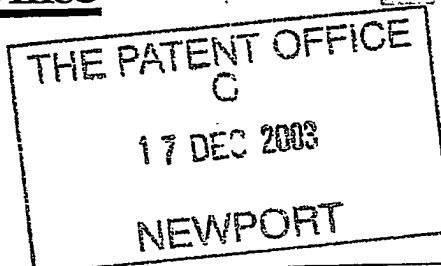
Stephen Hordley

Dated 28 September 2004

BEST AVAILABLE COPY

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)



The Patent Office

Cardiff Road
Newport
South Wales
NP9 1RH

1. Your reference

SN141

17DEC03 E860043-1 D02914
P01/7700 0.00-0329190.3 ACCOUNT CHA

2. Patent application number

(The Patent Office will fill in this part)

0329190.3

3. Full name, address and postcode of the or of each applicant (underline all surnames)

DOW CORNING CORPORATION
Midland
Michigan 48611
USA

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

USA/Michigan

8506040002

4. Title of the invention

FOAM CONTROL COMPOSITIONS

5. Name of your agent (if you have one)

P V Davies

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

DOW CORNING LIMITED
Intellectual Property Department
Cardiff Road
Barry
CF63 2YL

Patents ADP number (if you know it)

414037001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number
(if you know it)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

Yes

- a) any applicant named in part 3 is not an inventor, or
 - b) there is an inventor who is not named as an applicant, or
 - c) any named applicant is a corporate body.
- See note (d))

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description

16

Claim(s)

4

Abstract

Drawing(s)

Rm

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

1

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

1 fee sheet

11.

I/We request the grant of a patent on the basis of this application.

P V Davies

Signature

P V Davies

Date

16/12/2003

12. Name and daytime telephone number of person to contact in the United Kingdom

P V Davies 01446 723505

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

FOAM CONTROL COMPOSITIONS

[0001] This invention is concerned with foam control compositions for use in aqueous compositions which are liable to foam.

5

[0002] In many aqueous systems which are used e.g. in food processes, textile dyeing, paper production, sewage treatment and cleaning applications; the production of foam needs to be controlled or prevented. It is important to keep the foam formation to an acceptable level when laundering is performed in automatic washing machines, especially front loading machines. Excessive foam would cause overflow of the washing liquor onto the floor as well as reduction in the efficiency of the laundering operation itself. The foam control compositions of the invention can be added to detergent compositions to inhibit excessive foaming when the detergent is used in washing, or to aqueous media in food processes, textile dyeing, paper production or sewage treatment which are likely to foam.

10

15

[0003] The most successful foam control agents, especially for detergents, are based on silicones. Examples are described in US-A-5767053, US-A-6521586 and US-A-5387364. US-A-6521587 describes a foam control agent, comprising an organopolysiloxane material having at least one silicon-bonded aralkyl substituent, a water-insoluble organic fluid, an organosilicon resin and a hydrophobic filler. It is preferred that the water-insoluble organic fluid is miscible with the organopolysiloxane fluid at the operating temperature of the foam control agent.

20

[0004] There has been some demand for foam control agents containing a reduced amount of organopolysiloxane. US-A-5693256 describes a foam control agent comprising 100 parts by weight of a water-insoluble organic liquid, from 0.1 to 20 parts by weight of a first hydrophobic filler and 0.1 to 20 parts by weight of a second hydrophobic filler, which may be a siloxane resin, said hydrophobic fillers being insoluble in the water-insoluble organic liquid. EP-B-687724 describes a foam control agent comprising 100 parts by weight of a water-insoluble organic liquid, from 0.1 to 20 parts by weight of a hydrophobic filler that is insoluble in the organic liquid and 0.1 to 20 parts by weight of an organosilicon resin that is at least partially soluble in the organic liquid.

25

30

[0005] A foam control composition according to the invention comprises a liquid polymer of an unsaturated hydrocarbon, a branched siloxane resin, and a particulate filler which is insoluble in the liquid hydrocarbon polymer.

5

[0006] The preferred liquid hydrocarbon polymer is polyisobutene, also known as polyisobutylene or poly(2-methylpropene). Other liquid polymers of butene isomers can be used, for example a polymer of butene-1 and/or butene-2, as can other liquid hydrocarbon polymers such as polyisoprene. The liquid hydrocarbon polymer preferably has a molecular weight in the range 200 to 1500.

10

[0007] The branched siloxane resin and preferably consists of siloxane units of the formula $R'_aSiO_{4-a/2}$ wherein R' denotes a hydroxyl, hydrocarbon or hydrocarbonoxy group, and wherein a has an average value of from 0.5 to 2.4. It preferably consists of monovalent trihydrocarbonsiloxy (M) groups of the formula $R''_3SiO_{1/2}$ and tetrafunctional (Q) groups $SiO_{4/2}$ wherein R'' denotes a monovalent hydrocarbon group. The number ratio of M groups to Q groups is preferably in the range 0.4:1 to 2.5:1 (equivalent to a value of a in the formula $R'_aSiO_{4-a/2}$ of 0.86 to 2.15), more preferably 0.4:1 to 1.1:1 and most preferably 0.5:1 to 0.8:1 (equivalent to $a=1.0$ to $a=1.33$). The branched siloxane resin is preferably a solid at room temperature. The molecular weight of the resin can be increased by condensation, for example by heating in the presence of a base. The base can for example be an aqueous or alcoholic solution of potassium hydroxide or sodium hydroxide, e.g. a solution in methanol or propanol. A resin comprising M groups, trivalent $R''SiO_{3/2}$ (T) units and Q units can alternatively be used, or up to 20% of units in the branched siloxane resin can be divalent units $R''_2SiO_{2/2}$. The group R'' is preferably an alkyl group having 1 to 6 carbon atoms, for example methyl or ethyl, or can be phenyl. It is particularly preferred that at least 80%, most preferably substantially all, R'' groups present are methyl groups. The resin may be a trimethyl-capped resin. Other hydrocarbon groups may also be present, e.g. alkenyl groups present for example as dimethylvinylsilyl units, most preferably not exceeding 5% of all R'' groups. Silicon bonded hydroxyl groups and/or alkoxy, e.g. methoxy, groups may also be present.

15

20

25

30

[0008] The particulate filler is generally solid at 100°C and can for example be silica, preferably with a surface area as measured by BET measurement of at least 50 m²/g., titania, ground quartz, alumina, an aluminosilicate, an organic wax, e.g. polyethylene wax or microcrystalline wax, zinc oxide, magnesium oxide, a salt of an aliphatic carboxylic acids, a reaction product of an isocyanate with an amine, e.g. cyclohexylamine, or an high melting (above 100°C) alkyl amide such as ethylenebisstearamide or methylenebisstearamide. Mixtures of two or more of these can be used. The filler is preferably hydrophobic. Some of the fillers mentioned above are not hydrophobic in nature, but can be used if made hydrophobic. This could be done either in situ (i.e. when dispersed in the liquid hydrocarbon polymer), or by pre-treatment of the filler prior to mixing with the liquid hydrocarbon polymer. A preferred filler is silica which is made hydrophobic. Preferred silica materials are those which are prepared by heating, e.g. fumed silica, or precipitation. The silica filler may for example have an average particle size of 0.5 to 50µm, preferably 2 to 30 and most preferably 5 to 25µm. It can be made hydrophobic by treatment with a fatty acid, but is preferably done by the use of methyl substituted organosilicon materials such as dimethylsiloxane polymers which are end-blocked with silanol or silicon-bonded alkoxy groups, hexamethyldisilazane, hexamethyldisiloxane or organosilicon resins containing (CH₃)₃SiO_{1/2} groups. Hydrophobing is generally carried out at a temperature of at least 100°C. Mixtures of fillers can be used, for example a highly hydrophobic silica filler such as that sold under the Trade Mark 'Sipernat D10' can be used together with a partially hydrophobic silica such as that sold under the Trade Mark 'Aerosil R972'.

[0009] In one preferred embodiment of the invention, the branched siloxane resin is soluble in the liquid hydrocarbon polymer. For a branched siloxane resin consisting mainly of trimethylsiloxy groups and Q or T branching units, and a liquid hydrocarbon polymer such as polyisobutene, the resin is generally soluble in the liquid hydrocarbon polymer if the liquid hydrocarbon polymer has a molecular weight in the range 200 to 500. Branched siloxane resins in which residual hydroxyl groups are capped with methyl have clear solution compatibility with liquid hydrocarbon polymer of higher molecular weight in this range than resins that have not been capped. Polyisobutenes of higher molecular weight, for example 500 to 1500, can also be used to make effective foam control compositions although branched

siloxane resins are usually insoluble in these polyisobutenes of higher molecular weight. The liquid hydrocarbon polymer can be a blend of polymers; for example polyisobutene of Mw 750 can be a single polymer comprising molecules whose molecular weight is distributed about 750 or can be a blend of polyisobutenes characterized by the same viscosity.

5

[0010] The branched siloxane resin is preferably present at least 1%, more preferably at least 2% by weight based on the liquid hydrocarbon polymer, up to 40%, preferably up to 20%. For most antifoam uses it is preferred that the branched siloxane resin is present at 2 to 10% by weight based on the liquid hydrocarbon polymer.

10

[0011] The amount of hydrophobic filler in the foam control composition of the invention is preferably 0.5-50% by weight based on the polydiorganosiloxane fluid, more preferably from 1 up to 10 or 15% and most preferably 2 to 8%.

15 [0012] The blend of liquid hydrocarbon polymer, branched siloxane resin, and particulate filler is an effective foam control agent for many uses even when substantially free of polydiorganosiloxane fluid, particularly in controlling foam in aqueous media in food processes, textile dyeing, paper production or sewage treatment or in liquid detergent compositions.

20

[0013] The blend of liquid hydrocarbon polymer, branched siloxane resin, and particulate filler may further contain a substantially non-polar organic material of melting point 35 to 100°C which is at least partially miscible with the liquid hydrocarbon polymer. The non-polar organic material generally enhances the effectiveness of foam control achieved
25 by the blend. A preferred non-polar organic material of melting point 35 to 100°C comprises an organic polyol ester which is a polyol substantially fully esterified by carboxylate groups each having 7 to 36 carbon atoms. The polyol ester is preferably a glycerol triester or an ester of a higher polyol such as pentaerythritol or sorbitol, but can be a diester of a glycol such as ethylene glycol or propylene glycol, preferably with a fatty acid having at least 14 carbon
30 atoms, for example ethylene glycol distearate. Examples of preferred glycerol triesters are glycerol tripalmitate, which is particularly preferred, glycerol tristearate and glycerol triesters of saturated carboxylic acids having 20 or 22 carbon atoms such as that sold under the Trade

Mark 'Synchrowax HRC'. Alternative suitable polyol esters are esters of pentaerythritol such as pentaerythritol tetrabehenate and pentaerythritol tetrastearate. The polyol ester can advantageously contain fatty acids of different chain length, which is common in natural products. Most preferably the polyol ester is substantially fully esterified by carboxylate groups each having 14 to 22 carbon atoms. . By "substantially fully esterified" we mean that for a diol such as ethylene glycol or a triol such as glycerol, at least 90% and preferably at least 95% of the hydroxyl groups of the polyol are esterified. Higher polyols, particularly those such as pentaerythritol which show steric hindrance, may be "substantially fully esterified" when at least 70 or 75% of the hydroxyl groups of the polyol are esterified; for example pentaerythritol tristearate has the effect of a fully esterified polyol ester. The additive composition can comprise a mixture of polyol esters, for example a mixture containing carboxylate groups of different carbon chain length such as glyceryl tristearate and glyceryl tripalmitate, or glyceryl tristearate and Synchrowax HRC, or ethylene glycol distearate and Synchrowax HRC. Foam control compositions containing mixtures of two polyol esters in the additive composition may give greater foam control efficiency than compositions containing either polyol ester alone as the additive.

[0014] The non-polar organic material of melting point 35 to 100°C can alternatively be a hydrocarbon wax, for example it can comprise at least one paraffin wax, optionally blended with microcrystalline wax, for example the wax sold under the Trade Mark 'Cerozo'

[0015] The blend of liquid hydrocarbon polymer, branched siloxane resin, particulate filler and non-polar organic material of melting point 35 to 100°C may further contain a component which contains groups more polar than the groups present in the polyol ester non-polar organic material. The more polar group preferably contains an active hydrogen atom, that is one liable to undergo hydrogen bonding. Examples of more polar groups are unesterified -OH groups (alcohol or phenol groups), unesterified -COOH groups, amide groups or amino groups. The more polar component may have a melting point of at least 35°C, for example in the range 45-110°C, or may have a lower melting point, for example it may be liquid provided that the mixture of the non-polar and more polar components has a melting point of at least 35°C. The more polar component is preferably miscible with the

polyol ester and may also be miscible with the liquid hydrocarbon polymer. Examples of more polar components are fatty alcohols, ethoxylated fatty alcohols, ethoxylated fatty acids, ethoxylated alkyl phenols and partial esters of polyols such as monoesters or diesters of glycerol and a carboxylic acid having 8 to 30 carbon atoms, for example glycerol

5 monostearate, sorbitan monostearate, glycerol monolaurate or glycerol distearate, and mixtures thereof, alkyl phenols having one or more alkyl substituent and preferably containing a total of 6 to 12 carbon atoms in the alkyl substituent or substituents attached to the phenol nucleus, for example octylphenol or nonylphenol or di(t-butyl)phenol, fatty acids having 8 to 36 carbon atoms, for example stearic acid, palmitic acid, behenic acid, oleic acid
10 and/or 12-hydroxystearic acid, monoamides of fatty acids having 12 to 36 carbon atoms, for example stearamide, or alkyl amines having 8 to 30 carbon atoms.

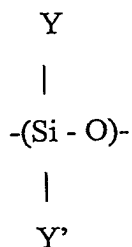
[0016] The substantially non-polar material of melting point 35 to 100°C and the more polar component can be present in weight ratio 5:95 to 95:5.

15 [0017] The additive composition, comprising the substantially non-polar material of melting point 35 to 100°C and optionally the more polar component, is preferably present in the foam control composition at 10-200% by weight based on the liquid hydrocarbon polymer, most preferably 20 up to 100 or 120%.

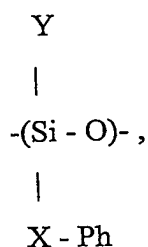
20 [0018] The organic compounds of melting point 35 to 100°C which have been described above as 'the more polar component' can alternatively be used as an additive to the blend of liquid hydrocarbon polymer, branched siloxane resin, particulate filler in place of the non-polar organic material, although this is less preferred.

25 [0019] The composition of the invention is also useful in enhancing the efficiency of foam control agents based on a polydiorganosiloxane fluid such as an organopolysiloxane material having at least one silicon-bonded aralkyl substituent. Thus according to another aspect of the invention a foam control composition comprises a liquid polymer of an
30 unsaturated hydrocarbon, a branched siloxane resin, and a particulate filler which is insoluble in the liquid hydrocarbon polymer, and additionally 10 to 100% by weight based on the liquid

hydrocarbon polymer of a polysiloxane fluid comprising at least 10% diorganosiloxane units of the formula



and up to 90% diorganosiloxane units of the formula



wherein X denotes a divalent aliphatic organic group bonded to silicon through a carbon atom; Ph denotes an aromatic group; Y denotes an alkyl group having 1 to 4 carbon atoms; and Y' denotes an aliphatic hydrocarbon group having 1 to 24 carbon atoms. Such a foam control composition has enhanced efficiency over known high performance foam control agents based on organopolysiloxane materials having at least one silicon-bonded aralkyl substituent, in particular in foaming systems containing a nonionic surfactant.

[0020] The liquid hydrocarbon polymer to be used in a foam control composition also containing a polysiloxane fluid preferably has a molecular weight in the range 500 to 1500. Liquid hydrocarbon polymers such as polyisobutene having a molecular weight of 750 or above are usually immiscible with polysiloxane fluids, particularly polydiorganosiloxanes having at least one silicon-bonded aralkyl substituent. We have found, surprisingly, that partial substitution of a polydiorganosiloxane by an immiscible polyisobutene oil such as that sold under the Trade Mark 'Glissopal 1000' (molecular weight 1000) leads to a large increase in defoaming persistence in certain nonionic surfactant solutions. Liquid hydrocarbon polymers such as polyisobutene of somewhat lower molecular weight, for example 350 to 550, can also be effective in improving defoaming persistence.

[0021] The diorganosiloxane units containing a -X-Ph group preferably comprise 5 to 40%, of the diorganosiloxane units in the fluid of the formula



and up to 90% diorganosiloxane units of the formula



The group X is preferably a divalent alkylene group having from 2 to 10 carbon atoms, most preferably 2 to 4 carbon atoms, but can alternatively contain an ether linkage between two alkylene groups or between an alkylene group and -Ph, or can contain an ester linkage. Ph is most preferably a phenyl group, but may be substituted for example by one or more methyl, methoxy, hydroxy or chloro group, or two substituents R may together form a divalent alkylene group, or may together form an aromatic ring, resulting in conjunction with the Ph group in e.g. a naphthalene group. A particularly preferred X-Ph group is 2-phenylpropyl - CH₂-CH(CH₃)-C₆H₅. The group Y' preferably has 1 to 18, most preferably 2 to 16, carbon atoms, for example ethyl, methyl, propyl, isobutyl or hexyl. Mixtures of different groups Y' can be present, for example a mixture of dodecyl and tetradecyl. Mixtures of alkyl groups Y' can be used. Other groups may be present, for example haloalkyl groups such as chloropropyl, acyloxyalkyl or alkoxyalkyl groups or aromatic groups such as phenyl bonded direct to Si. The polysiloxane fluid containing -X-Ph groups may be a substantially linear siloxane polymer or may have some branching, for example branching in the siloxane chain by the presence of some tri-functional siloxane units, or branching by a multivalent, e.g. divalent or trivalent, organic or silicon-organic moiety linking polymer chains, as described in US-A-6521587.

[0022] The foam control compositions according to the invention may be made by combining the liquid hydrocarbon polymer, the hydrophobic filler and the branched siloxane resin in any convenient way. The liquid hydrocarbon polymer, the hydrophobic filler and the organosilicon resin are preferably mixed together under shear. Where the filler needs to be made hydrophobic in situ, the manufacturing process includes a heating stage, preferably under reduced pressure, in which the filler and the treating agent are mixed together in part or all of the liquid hydrocarbon polymer, in the presence of a suitable catalyst if required.

[0023] The foam control composition of the present invention may be supported on a particulate carrier, particularly when the composition is to be used in a powdered product such as a detergent powder. Examples of carriers and/or supports are zeolites, for example Zeolite A or Zeolite X; other aluminosilicates or silicates, for example magnesium silicate, phosphates, for example powdered or granular sodium tripolyphosphate, sodium sulphate, sodium carbonate, for example anhydrous sodium carbonate or sodium carbonate monohydrate, sodium perborate, a cellulose derivative such as sodium carboxymethylcellulose, granulated starch, clay, sodium citrate, sodium acetate, sodium bicarbonate, sodium sesquicarbonate and native starch. The liquid hydrocarbon polymer containing the hydrophobic filler and the branched siloxane resin is preferably deposited on the carrier particles in non-aqueous liquid form, for example a temperature in the range 40-100°C. In an alternative process, the liquid hydrocarbon polymer, the hydrophobic filler and the branched siloxane resin and the non-polar additive if present are emulsified in water and the resulting aqueous emulsion is deposited on the carrier particles. The supported foam control composition is preferably made by an agglomeration process in which the foam control composition is sprayed onto the carrier particles while agitating the particles. The particles are preferably agitated in a high shear mixer through which the particles pass continuously. In one preferred process, the particles are agitated in a vertical, continuous high shear mixer in which the foam control composition is sprayed onto the particles. One example of such a mixer is a Flexomix mixer supplied by Hosokawa Schugi.

[0024] The supported foam control composition may additionally include a water-soluble or water-dispersible binder to improve the stability of the particles. Examples of binders are polycarboxylates, for example polyacrylic acid or a partial sodium salt thereof or a

copolymer of acrylic acid, for example a copolymer with maleic anhydride, polyoxyalkylene polymers such as polyethylene glycol, which can be applied molten or as an aqueous solution and spray dried, reaction products of tallow alcohol and ethylene oxide, or cellulose ethers, particularly water-soluble or water-swellaable cellulose ethers such as sodium

5 carboxymethylcellulose, or sugar syrup binders. The water-soluble or water-dispersible binder can be mixed with the foam control composition before being deposited on the carrier, but preferably is separately deposited on the carrier particles. In one preferred procedure the foam control composition is deposited on the carrier particles as a non-aqueous liquid at a

10 same time or subsequently, or at both times, deposited on the carrier from a separate feed as an aqueous solution or dispersion.

[0025] The supported foam control composition may optionally contain a surfactant to aid dispersion of the foam control composition in the binder and/or to help in controlling

15 the "foam profile", that is in ensuring that some foam is visible throughout the wash without overfoaming. Examples of surfactants include silicone glycols, or fatty alcohol ether sulphate or linear alkylbenzene sulphonate, which may be preferred with a polyacrylic acid binder. The surfactant can be added to the foam control composition undiluted before the silicone is deposited on the carrier, or the surfactant can be added to the binder and deposited as an

20 aqueous emulsion on the carrier.

[0026] The foam control composition can alternatively be provided in the form of an oil-in-water emulsion using any of the surfactants described in US-A-6521587. Alternatively the foam control agent can be provided as a water-dispersible composition in a water-

25 dispersible vehicle such as a silicone glycol or in another water-miscible liquid such as ethylene glycol, polyethylene glycol, propylene glycol, a copolymer of ethylene glycol and propylene glycol, an alcohol alkoxylate, an alkoxyalkanol or hydroxyalkyl ether or an alkylphenol alkoxylate.

30 [0027] The foam control agents according to this invention are useful for reducing or preventing foam formation in aqueous systems, including foam generated by detergent compositions during laundering and foam generated in such processes as paper making and

pulping processes, textile dyeing processes, cutting oil, coatings and other aqueous systems where surfactants may produce foam.

[0028] The following examples illustrate the invention. All parts and percentages are expressed by weight unless otherwise stated.

Example 1

[0029] 90% polyisobutene of molecular weight 550 was blended with 6% 'Sipernat D10' (Trade Mark) hydrophobic treated silica and 4% of a MQ siloxane resin having a ratio of M groups to Q groups of 0.65 to form Antifoam Composition A. The siloxane resin was dispersed in, but did not dissolve in, the polyisobutene.

[0030] Antifoam Composition A was deposited onto a sugar carrier to produce a supported foam control composition.

Example 2

[0031] 65% Antifoam Composition A was blended with 35% 'Synchrowax HRC' to form Antifoam Composition B.

Example 3

[0032] 80% polyisobutene of molecular weight 550 was blended with 20% polyisobutene of molecular weight 1000. 90% of the resulting polyisobutene blend was blended with 6% Sipernat D10 and 4% of the MQ siloxane resin described in Example 1. 65% of the resulting composition was blended with 35% 'Synchrowax HRC' to form Antifoam Composition C.

Example 4

[0033] 50% polyisobutene of molecular weight 550 was blended with 50% polyisobutene of molecular weight 1000. 90% of the resulting polyisobutene blend was

blended with 6% Sipernat D10 and 4% of the MQ siloxane resin described in Example 1. 65% of the resulting composition was blended with 35% 'Synchrowax HRC' to form Antifoam Composition D.

5 Example 5

[0034] 65% Antifoam Composition A was blended with 28% 'Synchrowax HRC' and 7% octylphenol to form Antifoam Composition E.

10 Example 6

[0035] 65% Antifoam Composition A was blended with 35% glyceryl monostearate (90% pure) to form Antifoam Composition F

15 [0036] Antifoam Compositions B, C, D, E and F were each sprayed onto sodium carbonate powder in a granulating mixer to produce a supported foam control composition containing about 16% of the active foam control composition.

Comparative Tests

20

[0037] Supported Antifoam Compositions A, B, C, D, E and F and comparative foam control agents G1 to G3 were tested in a powder detergent formulation which comprised 327 parts by weight zeolite, 95 parts of a 55% aqueous solution of sodium dodecylbenzenesulphonate, 39 parts ethoxylated lauryl stearyl alcohol, 39 parts sodium sulphate, 125 parts sodium carbonate and 125 parts sodium perborate. The comparative foam control agents were commercially used supported foam control compositions based on polydiorganosiloxane fluids containing hydrophobic silica. G1 and G2 used a zeolite carrier and G3 used a starch carrier. Each supported foam control composition was used at about 1% by weight of the detergent powder (0.15% by weight active foam control compound based on detergent powder). The evaluation was made in a Miele 934 front loading washing machine, loaded with 16 cotton towels, 100g of the detergent formulation, 17 litres of water of 9 degree German hardness using a wash cycle of 65 minutes at 40°C or 95°C. The foam height was

30

measured every 5 minutes during the wash cycle and recorded, where the value indicated is the foam height in the washing machine, with 100% referring to the fact that the window of the machine was full of foam, 50%, that is was half full of foam. The results are described in Table 1 below.

Table 1

Composition	Carrier	% Dosage	% Active	Temp C	% Foam height with time (mins)	5	10	15	20	25	30	35	40	45	50	55	60	65
A	Sugar	0.15		40	0	0	0	0	10	10	20	20	40	40	50	60	60	60
B	Soda Ash	0.15	16.91	40	0	0	0	0	0	0	0	0	5	5	5	15	20	25
C	Soda Ash	0.15	16.44	40	0	60	50	60	50	50	50	40	50	50	50	50	50	50
D	Soda Ash	0.15	16.37	40	0	80	90	90	90	90	100	100	100	100	100	100	100	100
B	Soda Ash	0.15	16.91	95	0	0	0	0	0	0	0	0	0	0	0	0	10	20
C	Soda Ash	0.15	16.44	95	0	50	50	50	50	40	10	0	0	0	10	10	20	20
D	Soda Ash	0.15	16.37	95	0	90	90	100	100	90	60	0	0	0	20	10	10	10
E	Soda Ash	0.15	16.40	40	0	0	0	0	0	0	0	0	0	0	10	20	20	30
E	Soda Ash	0.15	16.40	95	0	0	0	0	0	0	0	0	0	10	20	30	30	40
F	Soda Ash	0.15	16.51	40	0	40	30	10	0	10	20	30	50	60	60	70	80	80
G1	Zeolite	0.15	11.50	40	0	5	30	50	70	90	100	100	100	100	100	100	100	100
G2	Zeolite	0.15	11.12	40	0	0	0	0	0	0	0	5	20	25	35	45	50	50
G3	Starch	0.15	14.00	40	0	0	0	0	0	0	0	0	0	5	10	20	30	35
F	Soda Ash	0.15	16.51	95	0	50	30	10	20	20	20	30	40	50	50	40	40	40
G1	Zeolite	0.15	11.50	95	0	0	40	60	90	90	100	100	100	100	100	100	100	100
G2	Zeolite	0.15	11.12	95	0	0	0	0	0	0	10	10	10	10	20	10	0	0
G3	Starch	0.15	14.00	95	0	0	0	0	0	0	0	50	100	100	100	100	100	100

[0038] The foam control compositions of the invention showed effective foam control at both 40°C and 95°C and were in some conditions more effective than the commercial comparative compositions.

5 Example 7

[0039] 86% of a polydiorganosiloxane fluid comprising methyl ethyl siloxane groups and methyl 2-phenylpropyl siloxane groups was blended with 2% 'Cabosil TS720' (Trade Mark) hydrophobic treated silica and 12% of the branched siloxane resin used in Example 1
10 to form a silicone foam control agent. 50% of the silicone foam control agent was blended with 50% polyisobutene of molecular weight 1000 to form a foam control composition of the invention containing polyisobutene, hydrophobic silica, branched siloxane resin and polydiorganosiloxane fluid.

15 [0040] The foam control composition was tested in 'Triton X-100' (Trade Mark) alkyl phenol ethoxylate surfactant. The silicone foam control agent is known to have a poor persistence in this surfactant. Testing was done in the following way: 100 ml of 1% aqueous surfactant solution are placed in a 250 ml bottle; 20µl of the antifoam is added with a micro-syringe and the bottle submitted to 15 s shake cycles (with a wrist-action shaker). After each
20 shake cycle, one monitors the time for the produced foam to collapse to 10% of the free volume above the solution. The foam collapse time is plotted versus the number of shake cycles to show the evolution of defoaming activity with time. When the collapse time reaches 120 s we consider that the antifoam is deactivated.

25 [0041] The silicone foam control agent was deactivated after 29 shake cycles. The foam control composition of the invention showed a foam collapse time of below 70 seconds even after 40 cycles. The polyisobutene alone showed no antifoam activity (no foam collapse).

Examples 8 and 9

5 [0042] Foam control compositions of the invention were prepared by blending 20% of the silicone foam control agent of Example 7 with 80% polyisobutene of molecular weight 1000 (Example 8) or 10% of the silicone foam control agent of Example 7 with 90% polyisobutene of molecular weight 1000 (Example 9)

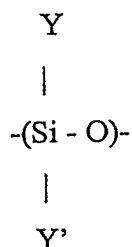
10 [0043] When the foam control compositions of Examples 8 and 9 were tested in the shake cycle test, they showed an even more persistent effect than the composition of Example 8. The foam collapse time stayed below 80 seconds for 52 shake cycles (Example 8) and 54 shake cycles (Example 9).

CLAIMS

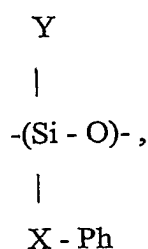
1. A foam control composition comprising a liquid polymer of an unsaturated hydrocarbon, a branched siloxane resin, and a particulate filler which is insoluble in the liquid hydrocarbon polymer.
2. A foam control composition according to Claim 1, characterised in that the liquid hydrocarbon polymer is polyisobutene.
3. A foam control composition according to Claim 1 or Claim 2, characterised in that the liquid hydrocarbon polymer has a molecular weight in the range 200 to 1500.
4. A foam control composition according to Claim 3, characterised in that the liquid hydrocarbon polymer has a molecular weight in the range 200 to 500 and in that the branched siloxane resin is soluble in the liquid hydrocarbon polymer.
5. A foam control composition according to any of Claims 1 to 4, characterised in that the branched siloxane resin consists of monovalent trihydrocarbonsiloxy (M) groups of the formula $R''_3SiO_{1/2}$ and tetrafunctional (Q) groups $SiO_{4/2}$ wherein R'' denotes an alkyl group and the number ratio of M groups to Q groups is in the range 0.4:1 to 1.1:1.
6. A foam control composition according to any of Claims 1 to 5, characterised in that the branched siloxane resin is present at 1 to 40% by weight based on the liquid hydrocarbon polymer.
7. A foam control composition according to Claim 6, characterised in that the branched siloxane resin is present at 2 to 10% by weight based on the liquid hydrocarbon polymer.

8. A foam control composition according to any of Claims 1 to 7, characterised in that the particulate filler is a silica filler with an average particle size of from 0.5 to 30 μ m.
9. A foam control composition according to any of Claims 1 to 8, characterised in that the particulate filler is present at 2 to 10% by weight based on the liquid hydrocarbon polymer.
10. A foam control composition according to any of Claims 1 to 9 which is substantially free of polydiorganosiloxane fluid.
11. A foam control composition according to any of Claims 1 to 10 which further contains a substantially non-polar organic material of melting point 35 to 100°C which is at least partially miscible in its molten state with the liquid hydrocarbon polymer.
12. A foam control composition according to Claim 11, characterised in that the substantially non-polar material of melting point 35 to 100°C comprises an organic polyol ester which is a polyol substantially fully esterified by carboxylate groups each having 7 to 36 carbon atoms.
13. A foam control composition according to Claim 11 or Claim 12, characterised in that the substantially non-polar material of melting point 35 to 100°C comprises 5-95 parts by weight of the organic polyol ester and 5-95 parts by weight of a component which is miscible in its molten state with the organic polyol ester and is more polar than the organic polyol ester component (A), at least one of the organic polyol ester and the more polar component being miscible with the liquid hydrocarbon polymer.
14. A foam control composition according to any of Claims 1 to 9 which additionally comprises 10 to 100% by weight based on the liquid hydrocarbon

polymer of a polysiloxane fluid comprising at least 10% diorganosiloxane units of the formula



and up to 90% diorganosiloxane units of the formula



wherein X denotes a divalent aliphatic organic group bonded to silicon through a carbon atom; Ph denotes an aromatic group; Y denotes an alkyl group having 1 to 4 carbon atoms; and Y' denotes an aliphatic hydrocarbon group having 1 to 24 carbon atoms.

15. A foam control composition according to Claim 14, characterised in that the liquid hydrocarbon polymer has a molecular weight in the range 750 to 1500 and in that the polysiloxane fluid is insoluble in the liquid hydrocarbon polymer.
16. A foam control agent according to any of Claims 1 to 15, which is provided in the form of an oil-in-water emulsion.
17. A water-dispersible foam control composition comprising a foam control agent according to any of Claims 1 to 15 dispersed in a water-dispersible carrier.
18. A granulated foam control agent comprising a foam control composition according to any of Claims 1 to 17 supported on a particulate carrier.

19. A granulated foam control agent according to Claim 18, characterized in that a water-soluble or water-dispersible binder is also deposited on the carrier particles.

GGA

PCT/US2004/041372



This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record.

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☐ **BLACK BORDERS**

☒ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**

☒ **FADED TEXT OR DRAWING**

☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**

☒ **SKEWED/SLANTED IMAGES**

☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**

☐ **GRAY SCALE DOCUMENTS**

☒ **LINES OR MARKS ON ORIGINAL DOCUMENT**

☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**

☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.